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Particle Behavior in
Solid Propellant Rocket
Motors and Plumes

by

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ABSTRACT

The particle size distribution inside the combustion chamber and the changes that occurred across the exhaust nozzle were measured in a subscale solid propellant rocket motor with a 2% aluminized end-burning propellant grain and a highly underexpanded nozzle. A combination of diagnostic techniques were used. Size distributions in the exhaust plume were determined by a Single Particle Counter, a Malvern 2600 ensemble particle sizer, and by Scanning Electron Microscope (SEM) examinations of particles collected on a timed exposure impact probe. Size distributions inside the combustion chamber were determined by Malvern 2600 measurements through windows at the nozzle entrance, SEM examinations of particles collected from the nozzle entrance wall, and exhaust plume measurements of a helium quenched motor. It was determined that agglomeration processes dominated in the flow from the center of the combustion chamber up to the nozzle entrance. Particle breakup processes dominated particle behavior from the nozzle entrance, through the nozzle, and into the exhaust.

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I. INTRODUCTION

Aluminum is added to many solid propellants to increase the delivered specific impulse (I_{sp}) and propellant density. Aluminum oxides formed by combustion can suppress transverse modes of combustion pressure oscillations, thus reducing the probability of combustion instability. However, the addition of aluminum to solid propellants is not without shortcomings. Within the motor, incomplete combustion of aluminum can lead to reduced combustion efficiency. The aluminum oxides formed by combustion can agglomerate into larger particles, typically from 50 μ m to 200 μ m in diameter (Price, 1984, p.480). These large particles can collect in the aft end of the motor, near submerged nozzle connections, to form slag. Nonuniform distributions of slag can cause flight instabilities. Particles that flow through the motor nozzle cannot expand with the gases. This two-phase flow can reduce the I_{sp} efficiency due to thermal and velocity lags between the particles and the gas. Aluminum in the propellant can also affect the exhaust plume signature. Particles exhausted by the rocket motor scatter ambient light. This scattering of light forms a visible exhaust called primary smoke. Particles in the exhaust can also scatter light that is radiated from the combustion chamber. These particles also emit radiation

proportional to approximately the fourth power of their surface temperature and proportional to their concentration.

Particles inside the motor can be helpful in damping pressure oscillations. The particles in the gas flowfield are 500-5000 times more dense than the gas (Price, 1984, p.755). As a result, the particles lag behind the gas in their response to pressure and velocity oscillations. This leads to damping of the wave as the gases oscillate back and forth past the particles. For a given gas coefficient of viscosity, particle density and combustion oscillation frequency, there exists an optimum particle radius to provide maximum damping (T'ien, 1983, p.824). Because of the effectiveness of particle damping, 0.05-3% of nonreactive particulate material of optimum radius is sometimes added to the propellant to suppress a specific combustion oscillation (Price, 1984, p.756). This nonreactive mass could be replaced with reactive aluminum if the aluminum oxide particles formed by combustion were the optimum size for damping. Unfortunately, this damping quality cannot be fully exploited without accurate knowledge of particle size distributions in the combustion chamber.

The determination of particle sizes inside the combustion chamber poses several problems. The particles are not easily accessible to intrusive diagnostic techniques, such as particle collection by probes, and few standard non-intrusive techniques have been successfully applied inside the

combustor. A non-intrusive technique has been used with limited success at the Naval Postgraduate School (Youngborg, 1990 and Brennan, 1992). Particle size distributions in the combustor were determined by sending a laser beam through windows on the sides of the combustion chamber, measuring the diffraction of the light, and converting to particle diameter by Fraunhofer diffraction theory. An interesting approach to the determination of particle sizes in the combustion chamber has also been discussed by Traineau and associates (Traineau, 1992). Traineau injected helium into the combustion chamber to quench/solidify aluminum and aluminum oxide particles formed during combustion. Maintaining the particulate in solid form allowed it to pass through the nozzle and into the exhaust unaltered. Identical tests were then conducted without the helium injection. The particle sizes at the nozzle exit were determined using the measurements of scattered laser light and scanning electron microscope examination of captured exhaust particles. A 30% helium injection mass flow rate close to the head-end of the combustion chamber (aft of the propellant grain) was used to provide an exhaust aluminum oxide particle size distribution that was assumed to be representative of the combustion chamber distribution. Traineau's technique allows the use of exhaust plume diagnostic methods to determine approximately the particle size distribution inside of the combustion

chamber, providing that the quench process does not induce particle breakup.

The techniques which have been most commonly used to determine particle sizes in rocket motor exhaust are based on electron microscope examination of collected samples, scattering or extinction of light from a multiple wavelength source, or scattering of light from a laser. Electron microscope examinations can yield size data as well as particle shape. However, thousands of particles must be measured to obtain an accurate size distribution. In addition, collecting particles with non-isokinetic, non-shock free impact probes may not give an accurate representation of size distribution. Since smaller particles can follow the gas around non-isokinetic impact probes and wall accumulation/shedding can produce larger agglomerates, electron microscope examinations of the collected exhaust can bias results toward larger particle sizes. In contrast, larger molten particles can break up passing through the probe, biasing the results to smaller particles. Multiple wavelength extinction measurements can be used to determine particle optical properties and diameter. However, particles must be spherical and smaller than about $1\mu\text{m}$ if UV or visible light is to be employed. This limits the practical use of multiple wavelength extinction measurements to only the edges of the exhaust plume. The scattering pattern of laser light from a particle can also be used to determine particle size.

These techniques are based either on the scattering from a collection of particles in a probe volume (ensemble technique) or on the scattering of light from a single particle at a time. The ensemble method has the advantage that all particles within the effective sample volume are measured, independent of position or velocity. However, ensemble measurements provide no spatial data and can give biased results due to density gradients in the flow (beam steering) and masking of larger particles from a concentration mismatch. Single particle counters can provide more detailed data about individual particles, such as spatial data (often including velocity), but are limited in particle number density and maximum velocity due to the requirement for recovering the scattered light from a single particle at a time. In general, single particle counters are often limited to a more narrow size range and lower concentration than ensemble measurements.

Data are needed to help clear up an existing controversy about the behavior of particulate matter in combustors and exhaust nozzles. Particle breakup/size reduction has been observed across the combustion chamber in subscale motors, but not explained with existing combustion/flow models. In the exhaust nozzle, particles can agglomerate (fast, small particles collide and stick to larger, slower particles), breakup due to high inertial forces, and/or accumulate and shed from wall surfaces. It is not clear which of these processes, if any, dominate the nozzle flow process.

The objective of this investigation was to obtain the particle size distribution inside the combustion chamber of a rocket motor and the changes in particle size distribution that occur across the exhaust nozzle. A small motor was used which was equipped with a helium injection system for quenching the reaction products as discussed by Traineau (Traineau, 1992). Several particle sizing diagnostic techniques were used. A Malvern (ensemble) particle sizer was used to measure the particle sizes at the nozzle entrance and the nozzle exit. In addition, a single particle counter (based on absolute intensity of scattered light) was used at the nozzle exit. Quenched and non-quenched tests were used to determine the particle size changes and to compare/validate the particle measurement diagnostic techniques. Particles collected from the chamber wall, nozzle wall, and from the surface of a short exposure impact probe in the exhaust plume were examined with a scanning electron microscope. These latter measurements were used for qualitative validation of the in-situ optical methods. More specifically the minimum, maximum, and most prevalent diameters were compared to those measured by the optical techniques.

II. EXPERIMENTAL APPARATUS

A. BACKGROUND

Two different three dimensional subscale rocket motors, a single particle counter sizing system, a Malvern 2600 ensemble type particle sizer, and a short exposure impact probe for collecting particles were used in the course of this experiment. Initial verification of the single particle counter's sizing ability was accomplished to increase confidence in the results achieved with the equipment (see Appendix A).

B. EQUIPMENT

1. Three Dimensional Subscale Motors

Two different solid propellant rocket motors were used to collect data. One of the rocket motors was configured to accommodate helium injection into the chamber for quenching of combustion products. The other motor did not have helium injector holes. Two rocket motors were required because particles would have been blown backwards into the helium injector holes in a non-quenched experiment. Each of these rocket motors was 2.00 inches in inside diameter and 9.25 inches long. A nitrogen-purged windowed section was attached to the end of these motors to allow measurements with the Malvern 2600 at the nozzle entrance. This windowed section

was 2.00 inches in diameter and 3.00 inches long. All experimental runs used this standard configuration to maintain a constant motor volume. The solid propellant was cut into cylindrical slabs approximately 1.98 inches in diameter and 1.00 inch thick. All experimental runs were conducted using a GAP/AP propellant with 2% aluminum and an end-burning grain. Detailed composition of the propellant may be found in Table I. Nitrogen purge gas was used across the quartz crystal windows when Malvern 2600 measurements were taken at the nozzle entrance. Otherwise, the windows were covered with stainless steel blanks and the nitrogen purge lines were capped. Ignition of the propellant was accomplished by using a BKNO_3 ignitor, which was fired by means of a nichrome filament energized by 12 volt DC power supply. The propellant was bound to the motor casing with a self vulcanizing silicone rubber compound (RTV). This not only bound the propellant to the casing but also inhibited burning from all surfaces except the exposed end of the grain. Minor differences between the two rocket motors are discussed below.

a. Non-quenched Motor

The rocket motor used for non-quenched experiments did not have helium injector holes. This motor was equipped with three ignitor ports from previous experiments. Two of these ignitor ports were plugged for this experiment. The ignitor port used was 2.36 inches from the head end of the

assembled motor. The nozzle had a throat diameter of 0.235 inches, exit diameter of 0.259 inches, giving an expansion ratio of 1.215. This nozzle was selected to provide a chamber pressure of 315 psi and exit velocity of approximately 1400 m/s (for compatibility with the single particle counter design limits). Figure 2.1 shows the assembled motor used for non-quenched experiments.

b. Quenched Motor

The rocket motor used for quenched experiments was similar to the non-quenched motor except that it had only one ignitor port 2.61 inches from the assembled head-end, and had 12 helium injector holes. The injector holes were radially spaced in two sets of six. The first set of 6 injector holes were 3.73 inches from the assembled head-end of the motor. The second set of 6 injector holes were 0.50 inches aft of, and staggered 30° from, the first set. Figure 2.2 shows the arrangement of the injector holes. The injector holes were 0.026 inches in diameter. These holes were designed to provide a subsonic helium flow rate of 0.0315 lb/s and a 0.87 Mach number for a chamber pressure of 315 psi and chamber temperature of 2007 K. Subsonic helium flow was desired to limit the breakage of aluminum/aluminum oxide particles by the helium injection (Traineau, 1992, p.6). A chamber temperature less than 2320 K was desired to solidify the molten aluminum oxide (Price, 1984, p.483). The nozzle used had a throat

diameter of 0.289 inches and an exit diameter of 0.309 inches, giving an expansion ratio of 1.143. This nozzle was selected to also provide a chamber pressure of 315 psi and an exit velocity of approximately 1700 m/s. Figure 2.3 shows the assembled motor used for quenched experiments.

2. Single Particle Counter

The single particle counter used was manufactured by Spectron Development Laboratories, Inc. for the HQ Ballistic Missile Office at Norton Air Force Base, California in July 1988. This system measures the size and velocity of particles in rocket plumes from 0.5 to 5 μm and up to 2000 m/s, respectively. This system was specifically designed to endure the harsh environment of rocket plumes. It is rated for 2000 K, 100 dB noise, and severe vibration. The maximum permissible particle density is $10^6/\text{cm}^3$. The maximum experiment time allowed is 4 seconds. Figure 2.4 shows a schematic of this optical system. A 4 Watt argon ion laser operating at $\lambda = 514.5 \text{ nm}$ was coupled to a single mode optical fiber by a x20 microscope objective. The output of this fiber was then collimated by a x10 microscope objective. The resultant laser beam was then passed through two cylindrical lenses to produce a laser sheet. A 230 mm achromatic lens focused the beam to produce an approximately cylindrical probe volume of 50 μm diameter (along the particle traverse direction) x 380 μm long. The rocket plume was directed through a 1 foot x 1 foot

opening between the transmitter and receiver housings. The probe volume was located 8.9 exit diameters aft of the nozzle exit plane. Light scattered by particles crossing the probe volume was collected by the receiver which was in the forward direction at approximately 4 from the transmitter. A combination of lenses in the receiver was used to collect and focus the scattered light onto a beam splitter. Scattered light focused on the reflective stripe of the beam splitter was reflected to a signal photomultiplier tube, while light missing the reflective stripe passed through the beam splitter to the mask photomultiplier tube. Particles whose scattered light reached the mask photomultiplier tube were rejected as being out of focus. The photomultiplier tube signals were sent to an electronics interphase box and then into a brassboard card installed in the IBM compatible personal computer for conditioning. The conditioned signals were then sent to a 200 MHz transient recorder where they were digitized and saved on hard disk. The digitized signals then went through a Gaussian fitting program to obtain the best fit between the measured and theoretical signals. The particle size was obtained from the peak amplitude and Mie scattering theory. The velocity was obtained from the value of the $1/e^2$ full width of the signal.

3. Malvern 2600 Particle Sizer

The Malvern 2600 particle sizer system was produced by Malvern Instruments of Malvern, England in 1985. The system uses a 2 mW helium-neon laser operating at $\lambda = 632.8$ nm. The laser beam passes from the transmitter and is scattered by particles on its way to the receiver. The light scattered by the particles and the unscattered remainder are incident onto a receiver lens, also known as a range lens. This range lens acts as a Fourier transform lens, forming the far field diffraction pattern of the scattered light at its focal plane. The scattered light is then collected over a range of solid angles by 31 concentric annular photodiode rings. The intensity of light collected by the annular rings is converted into particle sizes using Fraunhofer diffraction theory. The distribution of sizes is for the volume between the receiver and transmitter. Thus, the Malvern 2600 does not depend upon detection of single particles, but rather upon the net scattering of the collection of hundreds to tens of thousands of particles. This volumetric sizing technique is often called an ensemble measurement.

For this experiment a 100 mm range lens was used. This lens used forward scattered light with a maximum angle of approximately 9° . This provided a particle size range of 1.9-188 μm . An estimate of the volume of particles present with diameters between 0.5 and 1.9 μm is also provided. The vignetting distance associated with this lens is 133 mm. The

probe volume was located 8.9 exit diameters aft of the nozzle exit plane.

The accuracy of the Malvern is affected by several conditions. Beam steering from density gradients in the flow causes some difficulties. The correction for beam steering reduces the upper limit of particle size that can be accurately measured. Obscuration also affects the accuracy of the Malvern. Obscurations between 5-50% yield accurate sizes. Obscurations greater than 50% are subject to significant multiple scattering, which causes the Malvern to indicate particle sizes smaller than actual. Empirical corrections have been developed for high obscuration levels (Gülde, 1987, p.2).

4. Short Exposure Impact Probe

A stainless steel wedge was inserted into the plume for 0.5 seconds during rocket firing. The probe was located 21.8 exit diameters aft of the nozzle exit plane. The impact probe was 1 inch thick by 2 inches high by 1 inch wide, tapered down to 1/8 inch wide. Particles were collected by this probe and then qualitatively analyzed using a scanning electron microscope. The probe can be used uncovered, covered with double faced tape, or covered with a copper sheet. The double faced tape, acclaimed by Traineau to be the best surface for particle collection, proved to be difficult for mounting on SEM pedestals (Traineau, 1992, p.5). The best

results were found from the use of a copper sheet. The copper sheet did not require transfer of particles to the SEM pedestal and, therefore, gave a more representative measurement of collected particles.

III. EXPERIMENTAL PROCEDURE

A. SINGLE PARTICLE COUNTER VALIDATION

The Single Particle Counter had not been used before at the Naval Postgraduate School. Therefore, a validation of its capabilities was required to provide confidence in the results. The ideal validation process would have been the measurement of particles of known size and velocity. Unfortunately, a device of this type was not available for use. Instead, water droplets were produced by a six-jet atomizer and analyzed by both the Single Particle Counter and the Malvern MasterSizer system. The Single Particle Counter measured Sauter mean diameter was 0.687 μm and the Malvern MasterSizer measured Sauter mean diameter was 2.02 μm . These measurements were not in as close agreement as desired, but were considered close enough (see Appendix A) to continue with the experiments. The Single Particle Counter was operated in the Calibration mode for this validation due to the slow velocity of the atomized water.

B. PRE-FIRING PREPARATION

Prior to each experiment a dry run was conducted to ensure that the software used would control the sequence of events as planned. The pressure transducer was calibrated and then connected to the dead weight tester to provide a 100 psig

signal to simulate a motor firing. Actuator air was turned on for impact probe movement in Single Particle Counter experiments. The LABTECH NOTEBOOK program was executed and modified as required to achieve the desired sequence of events. Once the dry run was successful, the hardware was configured for the actual firing.

The propellant was cut to the desired diameter and length. The propellant was then coated with a self-vulcanizing silicone rubber compound (RTV) on non-burning surfaces for bonding and loaded into the head-end of a clean rocket motor. After at least a 24 hour curing period, the motor assembly was completed by installing the windowed section, nozzle, and burst disk assembly. The motor was then attached to the test stand and the pressure transducer was connected. For experiments which required motor windows, the windows were not installed until after a nitrogen purge was completed to ensure the nitrogen lines were completely dry. For experiments that did not require windows, stainless steel slugs were used in lieu of fused silica windows. The motor was positioned on the test stand to provide optimum alignment between the motor and the laser beam for data acquisition. The Single Particle Counter laser output was adjusted to provide 100-200 mW across the expected plume region. Nitrogen, helium, and actuator air were then set to desired pressures. A video camera was positioned for Single Particle Counter experiments to record plume events. The pre-assembled BKNO_3 ignitor was then

installed and connected to the 12 volt battery for power supply. A background reading for the laser data acquisition system was then recorded. Then the Single Particle Counter or the Malvern 2600 was configured to wait for an external trigger from LABTECH NOTEBOOK before commencing data acquisition. (Note: the external trigger for the Single Particle Counter failed on four experiments and was subsequently triggered manually.)

C. FIRING SEQUENCE

The video recorder was manually started for Single Particle Counter experiments. The firing sequence was started by executing the LABTECH NOTEBOOK program. Upon execution of this program, the nitrogen and helium gas solenoid valves opened to allow gases to flow as required by the experiment (gases were isolated from the solenoid valves for experiments that did not require their use). The ignitor was started by manually applying battery voltage to the nichrome wire embedded in the ignitor. The resulting current flow heated the nichrome wire and caused combustion of the BKNO_3 , which in turn ignited the propellant in the motor. When the chamber pressure reached approximately 100 psig, a timer was started. After a desired time delay, an external trigger was sent to the laser particle sizing system to commence data acquisition. The timing of this external trigger and the pressure time trace were recorded by LABTECH NOTEBOOK. (For three of the

four Single Particle Counter manually triggered data acquisition experiments, a signal was sent from the Single Particle Counter to LABTECH NOTEBOOK to mark the triggering event.) For Single Particle Counter experiments, the impact probe was inserted into the plume center after a desired time delay and removed from the plume 0.5 seconds later. After 15 seconds, the solenoid valves were shut to secure gas flow. The video recorder was then manually turned off. The motor was allowed to cool off and then disassembled and thoroughly cleaned in preparation for the next experiment.

Following the experiment, the data collected by LABTECH NOTEBOOK was manipulated to provide a pressure-time trace and data acquisition markers for correlation between chamber pressure and the sizing data collected. The data collected by the laser sizing system was then compared to the background recording and particle characteristics were determined. The video recording was studied to determine any motor leakage and for qualitative analysis of the plume.

D. PARTICLE COLLECTION MEASUREMENTS

After completion of the experiment, the particles collected by the impact probe and particles collected on the converging portion of the nozzle were sometimes transferred to SEM pedestals. These were then examined using a Scanning Electron Microscope (SEM) with the particle's composition identified by Energy Dispersive Xray (EDX) analysis. For

particles collected by the probe on copper sheets, there was no need to transfer particles to SEM pedestals. Instead, the copper sheets were trimmed, flattened, and then examined directly by the Scanning Electron Microscope. Photographs were taken from back-scattered electron images (BSEI).

IV. RESULTS AND DISCUSSION

A brief discussion of the results from experiments performed throughout November and early December 1992 at Naval Postgraduate School is presented here.

A. NON-QUENCHED EXPERIMENTS

A pictorial presentation of these results can be found in Figure 4.1 of Appendix C.

1. Single Particle Counter Plume Measurements

Four experiments were performed. The results from these four experiments were splined together to form one larger raw data base. This splined raw data was then converted to determine particle sizes. This splining of data provided the opportunity to obtain more statistically averaged results. The detected particles ranged in size from 0.67 to 1.24 μm in diameter. Of these, approximately 93% (number) were 0.86 - 1.24 μm . Additional Single Particle Counter results can be found in Table II. It should be noted that this instrument can only detect particles in the size range of 0.5 - 5 μm .

2. Malvern 2600 Plume Measurements

One experiment was performed. The Malvern collected data while the motor combustion chamber pressure was

increasing from 306 psig to 320 psig. The obscuration was 27%. Data on the inner eight diode rings had to be discarded due to beam steering from the large thermal gradients in the plume flowfield. This limited the maximum size that could be accurately determined (capturing all of the first Airy diffraction ring) to approximately 82 μm . There was a bimodal distribution of particle sizes with modes at less than 2 μm and approximately 3.5 μm (See Figure 4.2). The maximum detected particle size was 4.3 μm , by both the number and volume distributions. By number distribution, 87% were smaller than 2 μm . By volume distribution, 15% were smaller than 2 μm . The measured Sauter mean diameter (D_{32}) was 2.5 μm and the measured mass mean diameter (D_{43}) was 3.1 μm . With 87% of the particles (number) less than 2 μm and none larger than 4.3 μm , the Malvern results were in good agreement with the SPC measurements in the plume.

3. Plume Impact Probe SEM Examinations

Non-quenched particles were collected in the motor exhaust plume in four experiments. Particles were collected on a stainless steel probe for one experiment. The particles were washed from the probe and into a beaker with isopropyl alcohol. The sample was allowed to dry and then transferred to SEM pedestals. This technique was time consuming and yielded only a few particles for examination. All particles

observed were smaller than 5 μm in diameter. Most of the particles were submicron.

Particles were collected by double faced adhesive tape covering the stainless steel impact probe for one experiment. The particles were transferred to the SEM pedestals by touching the tape to a wet carbon painted pedestal. This technique also provided few particles for examination. These particles were all smaller than 5 μm and most were submicron. Some samples collected on adhesive tape were soaked in acetone, evaporated, and then the residue was transferred to SEM pedestals. This technique also provided very few particles for examination. All particles observed were submicron.

Particles were collected by copper sheets covering the impact probe for two non-quenched experiments. The copper sheet proved to be the best SEM technique. The relatively large difference in atomic number between the copper and the aluminum containing particles created an excellent contrast for back-scattered electron imaging analysis with the SEM. The particles observed ranged in sizes from 0.2 to 2.3 μm with the majority of particles being smaller than 0.5 μm (See Figures 4.3 and 4.4). Considering that the Malvern estimates particles only to a minimum of 0.5 μm and that this was also the lower limit of the SPC, the collected particle sizes were in good agreement. On the slanted sides of the impact target probe, the particles had piled on top of one another during

impact to form irregularly shaped patches. These patches varied greatly in size and shape. The largest patch was over 2 mm long and over 50 μm wide. The smallest patch was nearly round with a diameter 2.4 μm . On the front edge of the impact probe, the particles dug craters into the copper sheet and collected on the rim (See Figure 4.5).

4. Malvern 2600 Nozzle Entrance Measurements

One successful non-quenched experiment using the Malvern 2600 through the motor windows was achieved following two failed attempts due to inadequate nitrogen purge. The successful nitrogen purge used 2100 psig nitrogen manifold pressure and one 0.030 inch sonic choke. This was designed to provide a nitrogen flowrate which was 26% of the propellant mass flowrate. This nitrogen flow should only decrease the temperature down to approximately 2760 K according to chemical equilibrium calculations. This temperature was well above the melting temperature of aluminum oxide and therefore should have a negligible effect on particle size. The Malvern collected data while the combustion chamber pressure was increasing from 430 psig to 450 psig. The obscuration was 95%. The approximate volume concentration of particles in gas was 3×10^{-5} , based on the propellant burning rate and chamber pressure. With $D_{32} = 17 \mu\text{m}$, as measured by the Malvern, the obscuration should be only approximately 22%. Thus, it was apparent that some beam steering was present. Beam steering

deflects some of the central focused (unscattered) light from the pinhole in front of the diode. However, the beam steering was overpowered by the scattered light that reached the first few diode rings. The implication of this behavior was that the measured size distribution was not significantly affected by the "indicated" high obscuration. The maximum detected size was 84 μm , D_{32} was 17.3 μm , and D_{43} was 28.8 μm . From the number distribution, 65% of the particles were smaller than 2 μm and 98% were smaller than 11 μm . From the volume distribution, 0.2% were smaller than 2 μm , 13% were smaller than 11 μm , and 99% were smaller than 55 μm . The mode peaks of the distribution were smaller than 2 μm , 3.5 μm , 7 μm , 18 μm , and 43 μm (See Figure 4.6). Thus, most of the number of particles were smaller than 2 μm , whereas most of the mass was contained in particles larger than 10 μm .

5. Nozzle Wall SEM Examinations

Particles were scraped from the converging wall of the nozzle and deposited onto wet SEM pedestals. The maximum particle size observed was 40 μm . The smallest size observed was 0.5 μm . The particles were primarily distributed between four sizes: 0.7 μm , 2 μm , 6 μm , and 25 μm . Most particles were of the 0.7 μm and 2 μm sizes (See Figure 4.7). As displayed in Figure 4.8, the aluminum oxide structure of the larger particles was that of a cracked shell similar to that seen in other experiments (Price, 1984, p. 487 and Traineau,

1992, p. 11). The Malvern indicated that most of the particles (number) were smaller than 2 μm , in agreement with the collected particles. The Malvern also indicated a mode peak at 43 μm (i.e. a significant number of particles), in agreement with the maximum observed size from the SEM of 40 μm .

Each of the diagnostic techniques employed had different dynamic ranges. With this in mind, all of the results were in quite good agreement.

B. QUENCHED EXPERIMENTS

A pictorial presentation of these results can be found in Figure 4.9 of Appendix C.

1. Single Particle Counter Plume Measurements

Two experiments were performed. The results were splined together to improve statistical averaging of results. The detected particles ranged in size from 0.48 to 1.43 μm . Of these, approximately 86% were 0.67 - 1.05 μm . Additional results can be found in Table II.

2. Malvern 2600 Plume Measurements

One experiment was performed. The Malvern collected data while the chamber pressure was approximately level at 272 psig. The obscuration was 23%. Beam steering occurred as for the non-quenched measurements. This again limited the maximum measurable accurate size to approximately 82 μm . The maximum size detected was 30 μm , D_{32} was 3.2 μm , and D_{43} was 13.8 μm .

By number distribution, 95% were smaller than 2 μm and 100% were smaller than 6 μm . By volume distribution, 21% were smaller than 2 μm , 42% were smaller than 6 μm , and 100% were smaller than 31 μm . The mode peaks of the distribution were smaller than 2 μm , 2 μm , 4.5 μm , 12 μm , and 26 μm (See Figure 4.10). With 95% of the number of particles measured less than 2 μm , the Malvern and SPC results were in reasonably good agreement. The SPC could not detect the very few number of large particles due to both the dynamic range limitations and to the highly improbable event of a single large particle passing through the relatively small measurement volume.

3. Plume Impact Probe SEM Examinations

Quenched particles were collected on copper sheets in the exhaust plume for two experiments. The particles observed ranged in size from 0.3 to 2 μm with the majority being smaller than 0.5 μm (See Figure 4.11). This result was in good agreement with the Malvern and SPC measurements, although none of the few number of larger particles seen by the Malvern were observed. The particles piled on top of each other on impact as was observed in the non-quenched case. The craters formed on the front edge of the probe were not as well defined as had been observed for the non-quenched case (See Figure 4.12).

4. Malvern 2600 Nozzle Entrance Measurements

Two unsuccessful attempts were made to measure particle sizes through the windows of a quenched motor. One experiment was rejected because a Malvern sample taken approximately five minutes after the firing indicated that the windows had been fouled. The other experiment was rejected because the sample was taken during the rapid chamber pressure decrease at the tailoff of the motor firing. Further attempts were not made due to time constraints.

5. Nozzle Wall SEM Examinations

Particles were scraped from the converging wall of the nozzle and mounted on pedestals. The maximum size observed was 59 μm . The minimum size observed was 0.6 μm . The particles were primarily of five sizes: 0.7 μm , 2 μm , 7 μm , 25 μm , and 50 μm . Most of the particles were smaller than 2 μm . (See Figure 4.13) The aluminum oxide particles displayed cracked shells as was seen in the non-quenched case (See Figure 4.14). These aluminum oxide particles had a greater silicon content (from the inhibitor) than found in the non-quenched case. Also, there were smooth spherical masses of mostly silicon that had not been observed in the non-quenched case. The smooth masses in Figure 4.15 are composed of mostly silicon. Since the Malvern did not detect particles larger than 31 μm in the plume and the impact probe also saw only small particles, the implication was that the larger particles

may have not been quenched to solid form before passing through the nozzle.

C. COMPOSITE PICTURE OF PARTICLE BEHAVIOR

In this investigation, only a small amount of aluminum oxide was present (maximum of 3.8% by mass), the pressure averaged about 350 psi, there was significant residence time between the end-burning propellant grain surface and the nozzle entrance, and the plume measurements were made near the exit of a highly underexpanded (P_{exit} approximately 100 psi) nozzle. The results indicated that the motor quench probably did not solidify all of the larger particles before they passed through the exhaust nozzle. Assuming that the quench process did not shatter particles, the above data indicate that the smaller particles overtook and collided with the larger particles as they passed along the length of the combustor (D_{max} increased from 59 μm to 84 μm and the number of and volume of <2 μm particles decreased from 95% to 65% and 21% to 0.2%, respectively). In passing through the highly underexpanded nozzle, particle breakup dominated, with no particles larger than 5 μm observed.

The results obtained at the nozzle entrance for 350 psi were similar, yet somewhat different from those observed by Laredo and Netzer for 420 psi (Laredo and Netzer, 1992). Their results had a higher mass percentage of <2 μm particles (9% versus 0.2%) and a smaller maximum particle size (12 μm

versus 84 μm). Traineau reported (for a different propellant and operating conditions) approximately 10% of the mass $< 2 \mu\text{m}$, most particles smaller than 75 μm , and a maximum of 120 μm (Traineau, 1992). Although these results for the nozzle entrance vary somewhat for the different propellants and test conditions, they give a consistent picture. Most of the number of particles are smaller than 2 μm but most of the mass (greater than 90%) is contained in particles larger than 2 μm diameter. Also, most particles are smaller than 50 μm , although a few as large as 80 - 120 μm are present. At the nozzle exit with a highly underexpanded flow, the investigation indicated that particle breakup dominated over agglomeration within the nozzle and near plume region. Laredo and Netzer also indicated that breakup dominates, but showed that observable agglomeration also occurs (Laredo and Netzer, 1992). In Traineau's experiments, the optical technique (light scattering) indicated the smallest particles agglomerated and the larger particles shattered. However, based on the beam steering observed in the present investigation, Traineau's measured maximum plume size of 120 μm may have resulted from beam steering effects. (Traineau, 1992)

V. CONCLUSIONS AND RECOMMENDATIONS

The purpose of this investigation was to obtain the particle size distribution inside the combustion chamber of a rocket motor and the changes in particle size distribution that occur across the exhaust nozzle. Several particle sizing diagnostic techniques and the quenching of aluminum oxide particles were used successfully. The results obtained by the various diagnostic techniques were in good agreement when consideration was given to their respective size range limitations.

In-situ optical techniques can be used together with collected particles to determine the changes in particle sizes that occur from the combustion chamber to the nozzle entrance and into the exhaust plume. In the combustion chamber, there was a multimodal size distribution (from 0.5 - 60 μm) with most of the number of particles being smaller than 2 μm but most of the mass (approximately 80%) being in larger particles. As the particles passed along the combustor length to the nozzle entrance, many of the <2 μm particles collided and combined with larger particles to form a wider multimodal distribution (0.5 - 84 μm) with less than 1% of the mass contained in particles <2 μm . As the particles flowed through the nozzle and into the plume, the larger particles (>3.5 μm)

broke up into smaller particles and formed a bimodal distribution (<2 and $3.5 \mu\text{m}$). Therefore, it was concluded that the chamber process was dominated by an agglomeration mechanism and the nozzle process was dominated by a breakup mechanism.

The following recommendations are made for further experimentation in this area of research:

- Malvern 2600 experiments need to be performed successfully through the quenched motor windows.
- Further validation is needed for the Single Particle Counter using known particle sizes and velocities.
- A study of the effect of quench gas Mach number and mass flowrate should be performed to ensure that the quench gas is not the source of particle breakup and that the larger particles are indeed quenched.

APPENDIX A
SINGLE PARTICLE COUNTER VALIDATION

The Single Particle Counter developed by Spectron Development Laboratories had not been previously used at the United States Naval Postgraduate School. Therefore, a validation of this equipment's performance was required. The ideal validation using known particle sizes and velocities was not possible. Therefore, a different technique was used. A six-jet water atomizer was used to produce a fine mist of water droplets. These droplets were analyzed with both the Single Particle Counter and the Malvern MasterSizer. The Malvern MasterSizer has been used successfully since 1989 at the Naval Postgraduate School. Therefore, it was assumed that the analysis of water droplets by the Malvern MasterSizer would result in approximately the true particle sizes.

The mist was analyzed by the Single Particle Counter seven times. The Single Particle Counter detected particles in primarily three size ranges. Of the detected particles, 85.6% were 0.48 - 0.86 μm , 13.4% were 1.05 - 1.24 μm , and 1% were 2.67 μm in diameter. The velocity ranged from 16 - 50 m/s.

The mist was analyzed by the Malvern MasterSizer three times. The Malvern MasterSizer used a 100 mm lens which provided a size range of 0.2 - 180 μm . The Malvern

MasterSizer, using a model independent number distribution, detected particles in primarily four size ranges. All particles detected were smaller than 5.79 μm . Of the particles detected 53.6% were 0.2 - 0.48 μm , 12.4% were 0.48 - 0.59 μm , 29.7% were 0.59 - 1.52 μm , and the remaining 4.3% were 1.52 - 5.79 μm in diameter.

The results of both devices indicated mostly submicron particles. Note that the Malvern MasterSizer measured the majority of particles in a size range smaller than the capability of the Single Particle Counter. An examination of Malvern MasterSizer size ranges from 0.48 to 5.79 μm (to coincide with the Single Particle Counter's capabilities) reveals 91% were 0.48 - 1.52 μm . This was in good agreement with Single Particle Counter measurements of 99% sized 0.48 - 1.24 μm . Therefore, the Single Particle Counter was determined to function as specified in the user's manual. Future work with the Single Particle Counter should involve measurement of known size and velocity particles for a more precise validation.

APPENDIX B

TABLES

TABLE I

PROPELLANT COMPOSITION

	DD-2
AP (200 microns)	47.450%
AP (20 microns)	25.550%
GAP	14.670%
TEGDN	8.490%
Aluminum	3.490%
N - 100	0.490%
HDI	0.845%
Tepanol	0.490%
<p>Burning rate (in/sec) = $0.0592 \cdot (P_c)^{0.362}$ where P_c is in psia</p>	

TABLE II
SPC SPLINED PLUME RESULTS

	Non-Quenched	Quenched
Linear mean diameter (μm)	0.982	0.818
Sauter mean diameter (μm)	1.052	0.910
Maximum diameter (μm)	1.24	1.43
Minimum diameter (μm)	0.67	0.48
Prominent diameters (μm)	92.6% were 0.86 - 1.24	86.4% were 0.67 - 1.05
Linear mean velocity (m/s)	2177	1727
RMS velocity (m/s)	1489	1199

APPENDIX C

FIGURES

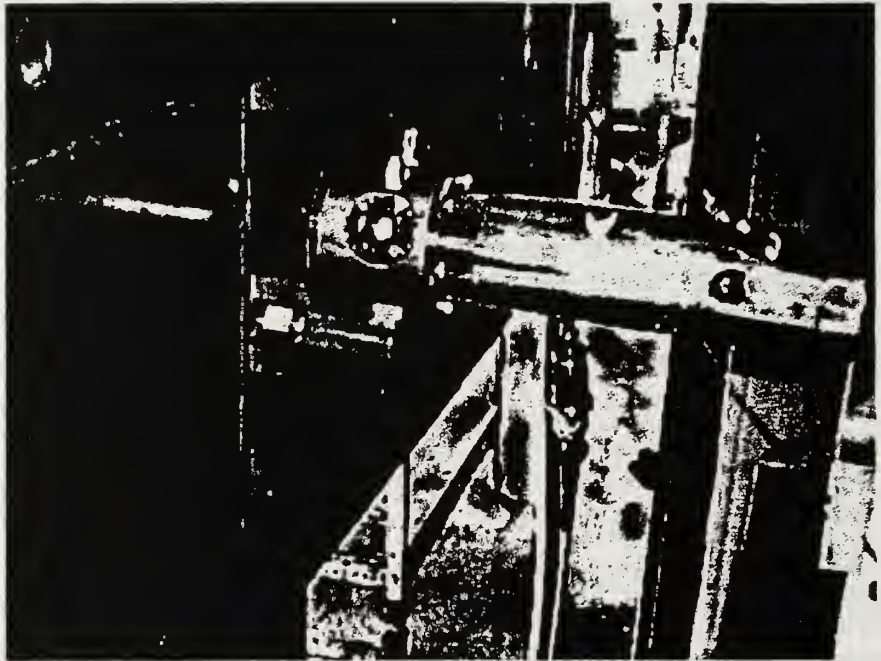


Figure 2.1 Non-Quenched Motor

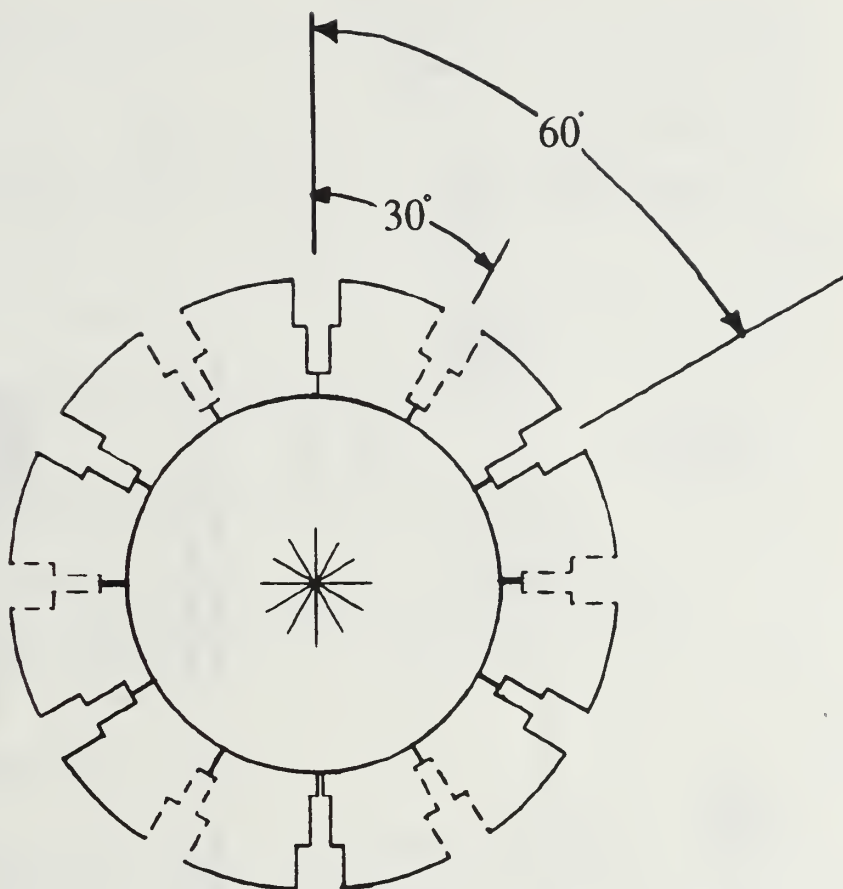


Figure 2.2 End View of Helium Injector Holes



Figure 2.3 Quenched Motor

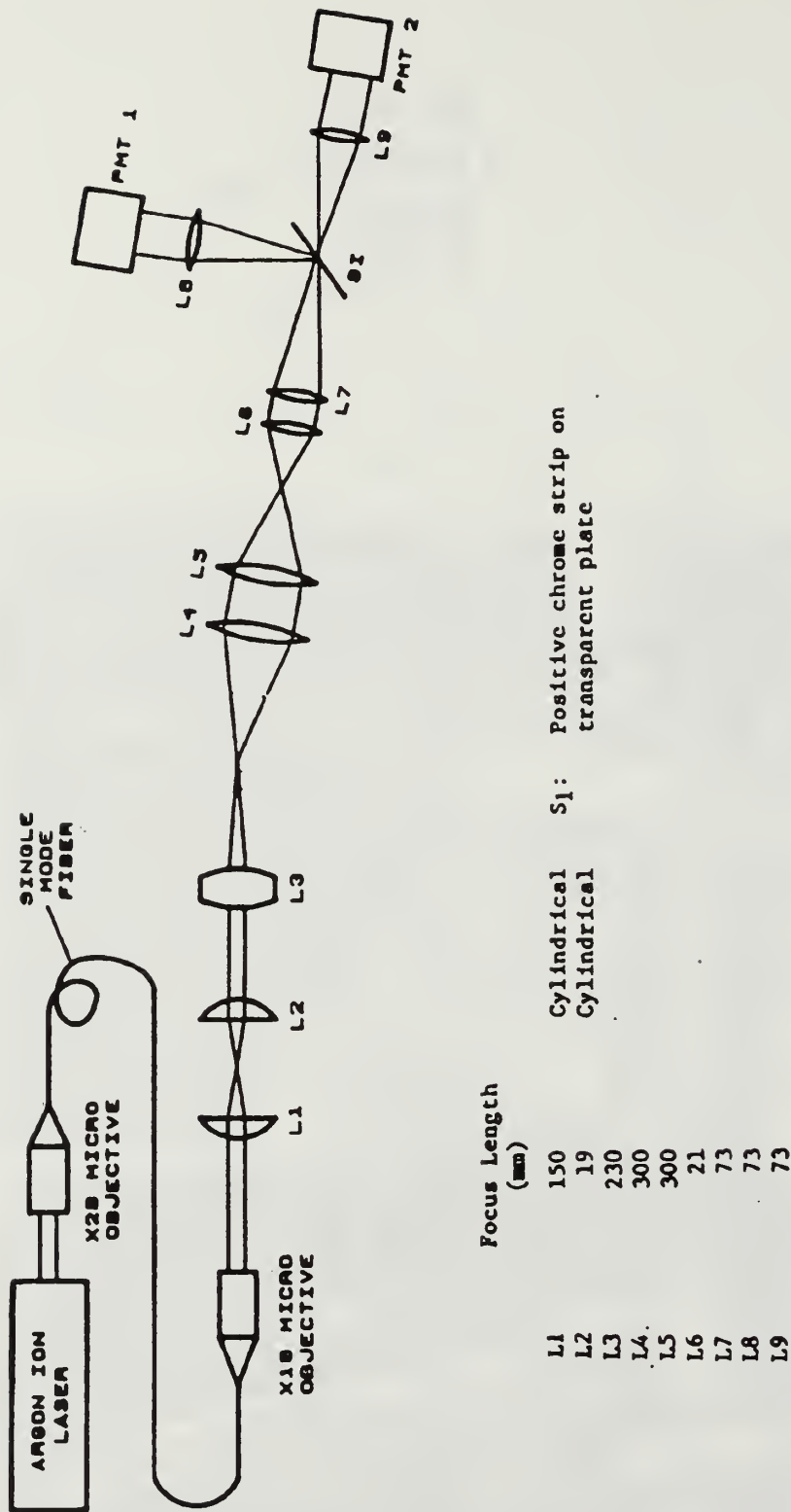


Figure 2.4 Single Particle Counter Optical Configuration
(SPC User's Manual, 1988)

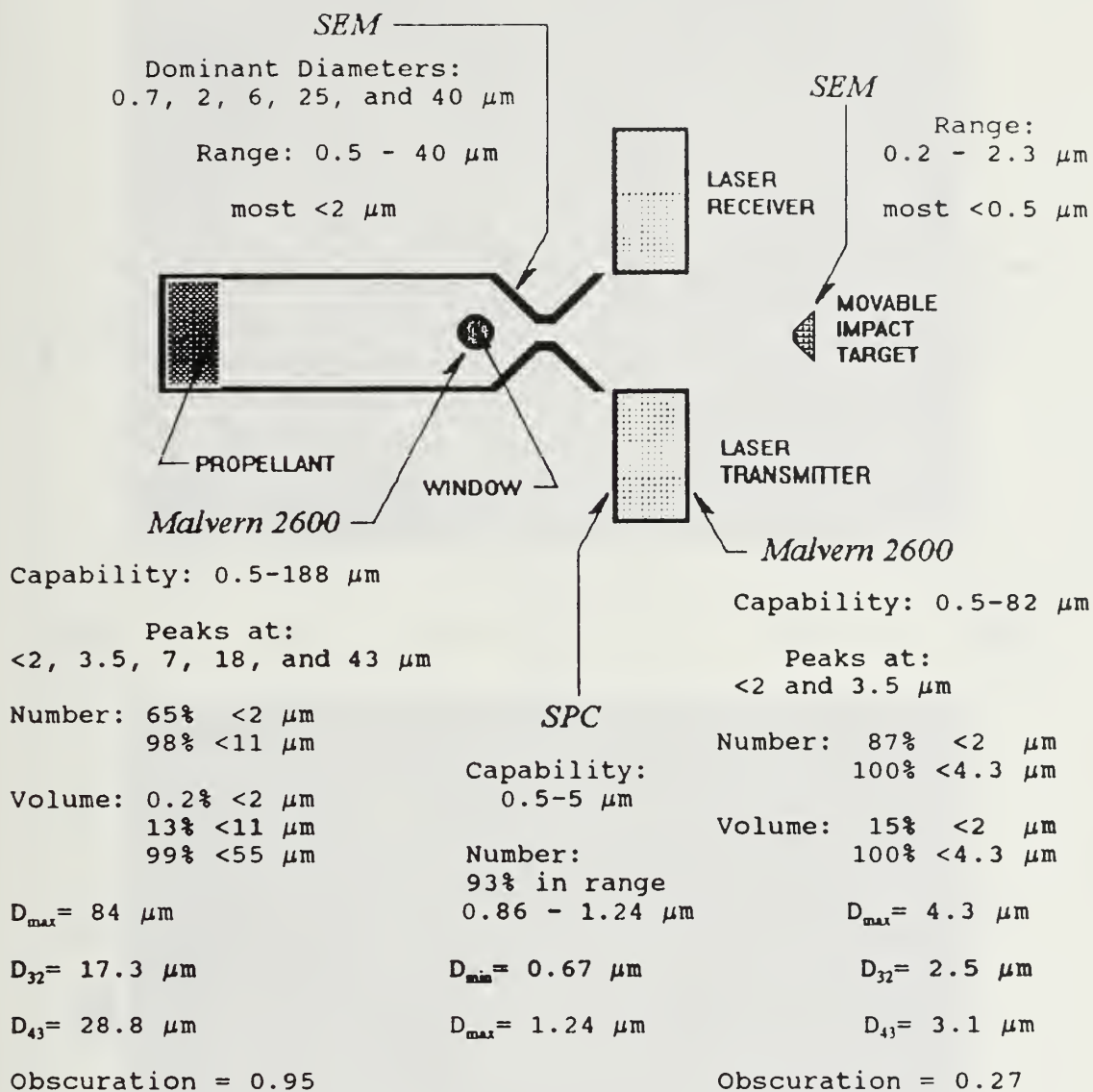


Figure 4.1 Non-Quenched Motor Results

MALVERN Series 2600 SB.0A Master Mode Mon 30 Nov 1992 11:46 am

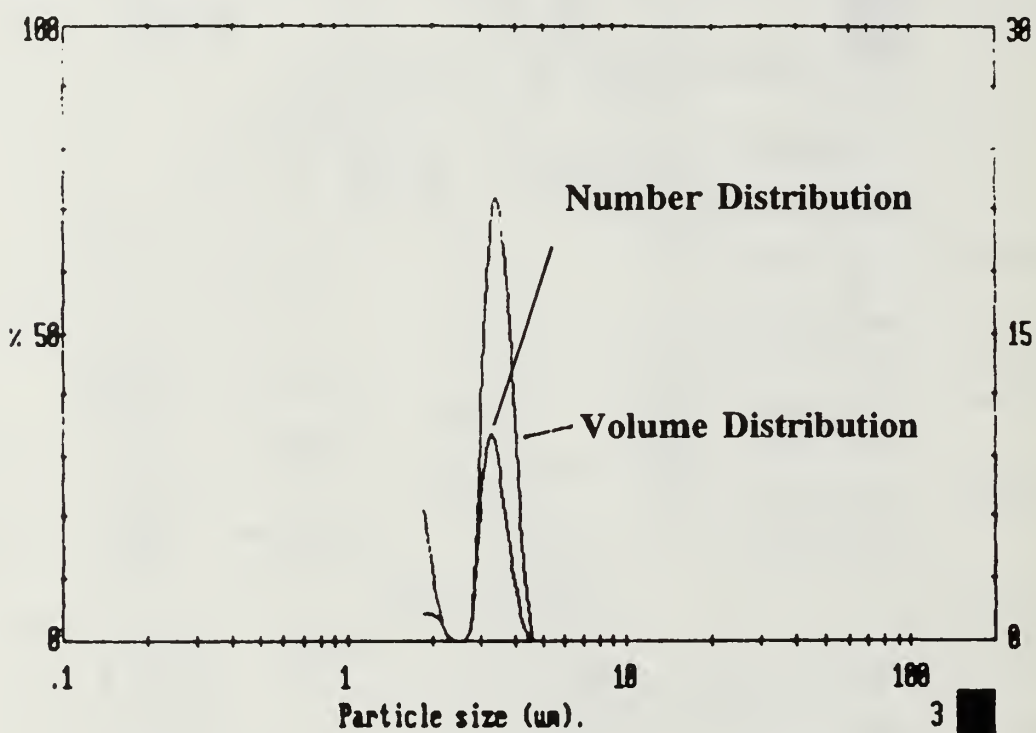


Figure 4.2 Non-Quenched Malvern 2600 Plume Results

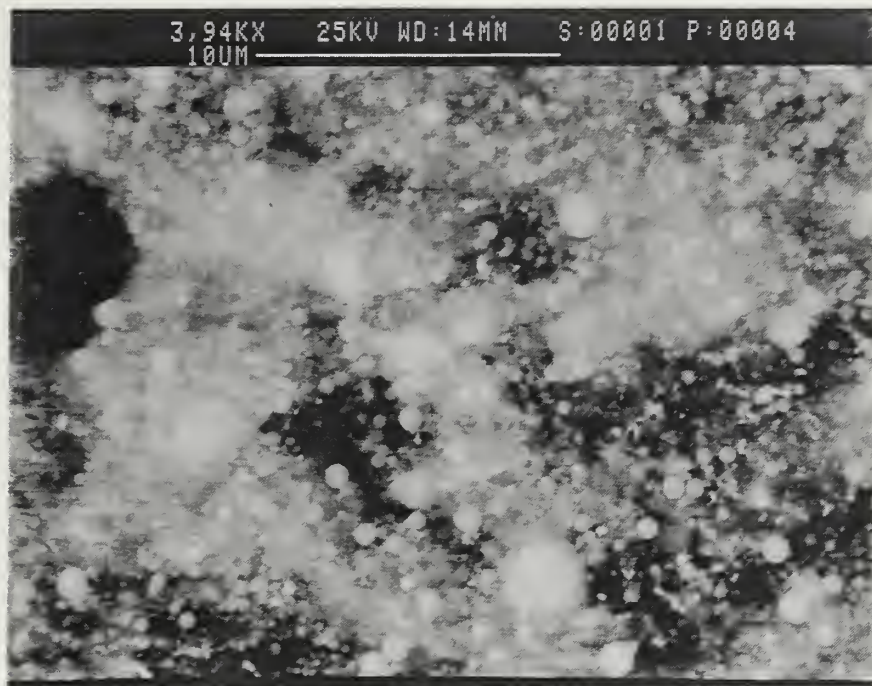


Figure 4.3 Non-Quenched Impact Probe Sample (3,94 KX)



Figure 4.4 Non-Quenched Impact Probe Sample (20,6 KX)

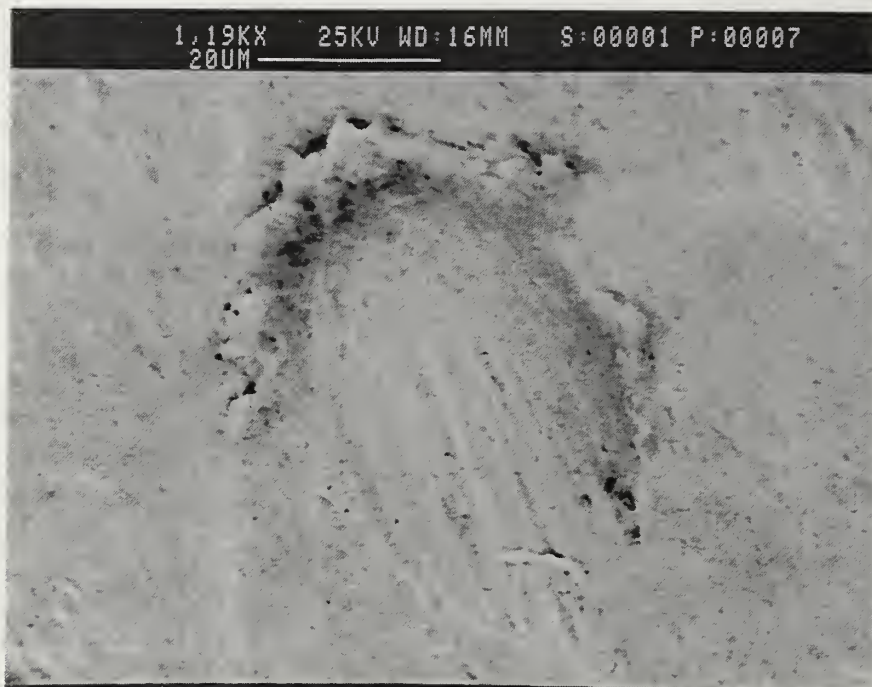


Figure 4.5 Non-Quenched Impact Probe Crater
MALVERN Series 2600 SE.00 Master Mode Wed 2 Dec 1992 3:49 pm

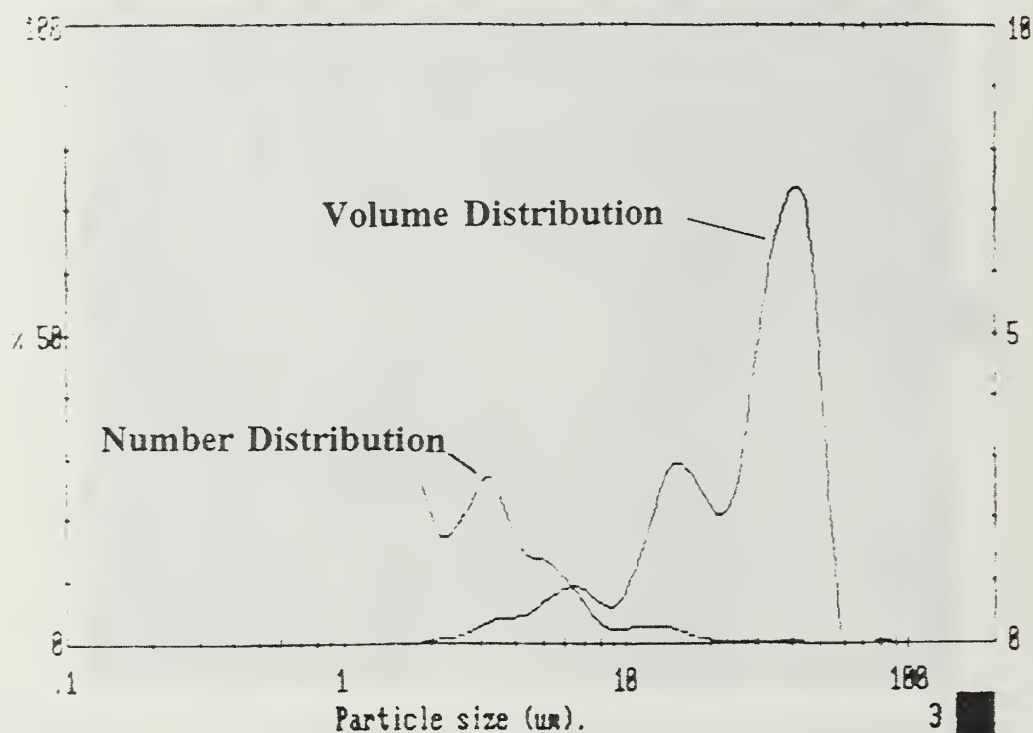


Figure 4.6 Non-Quenched Malvern Nozzle Entrance Results

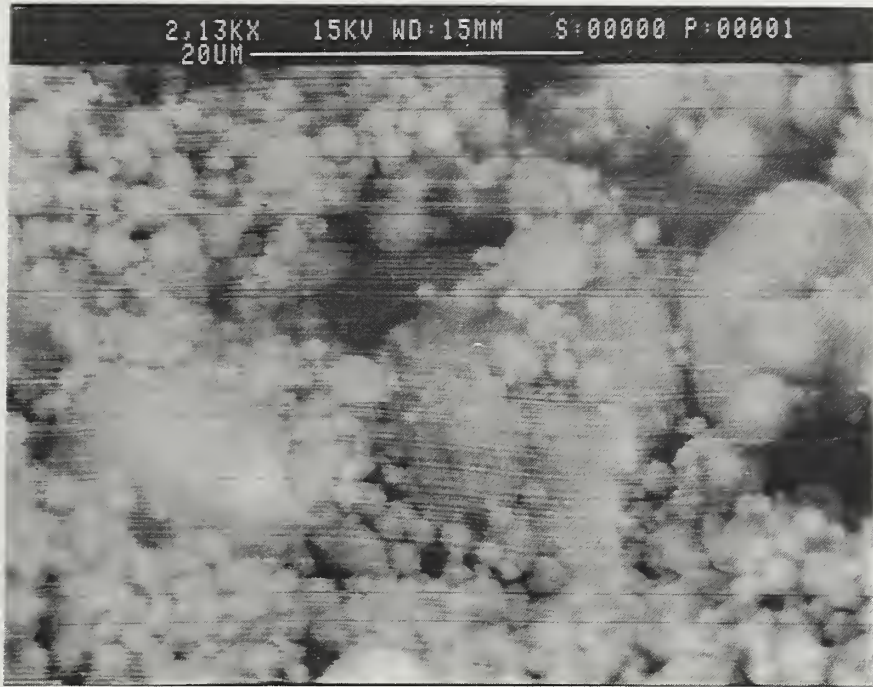


Figure 4.7 Non-Quenched Nozzle Sample

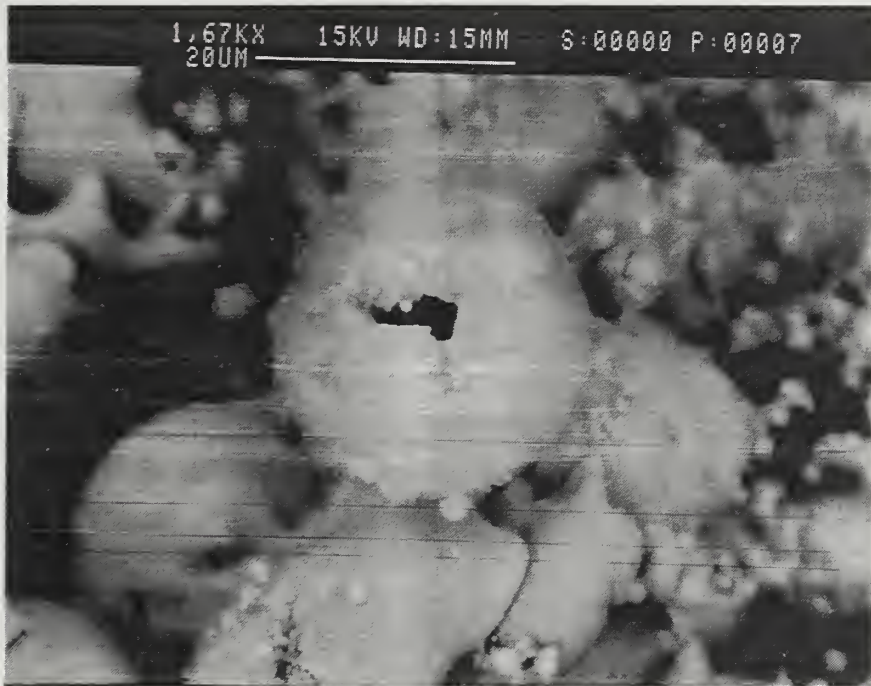


Figure 4.8 Non-Quenched Nozzle Sample Showing Cracked Shell

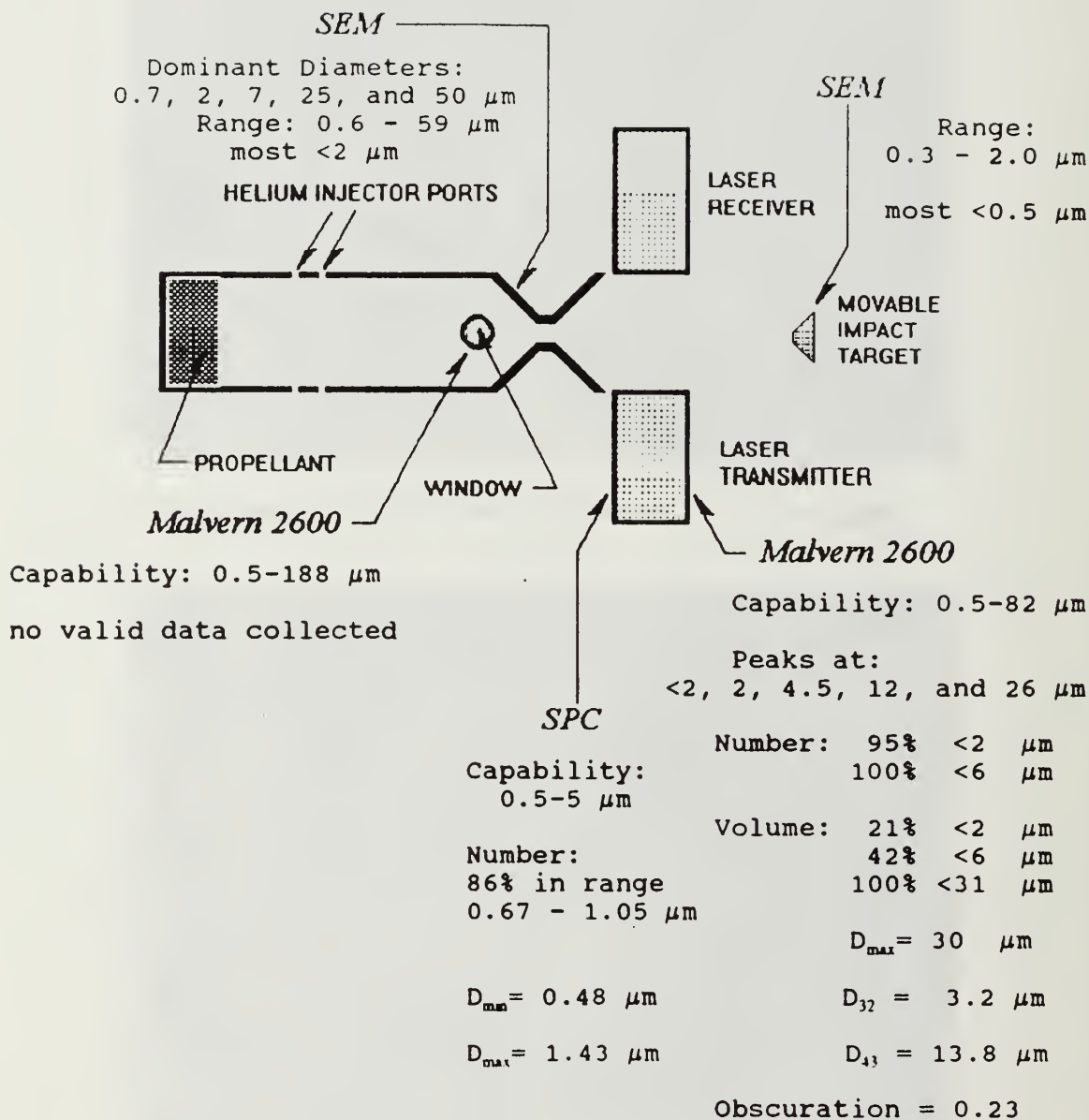


Figure 4.9 Quenched Motor Results

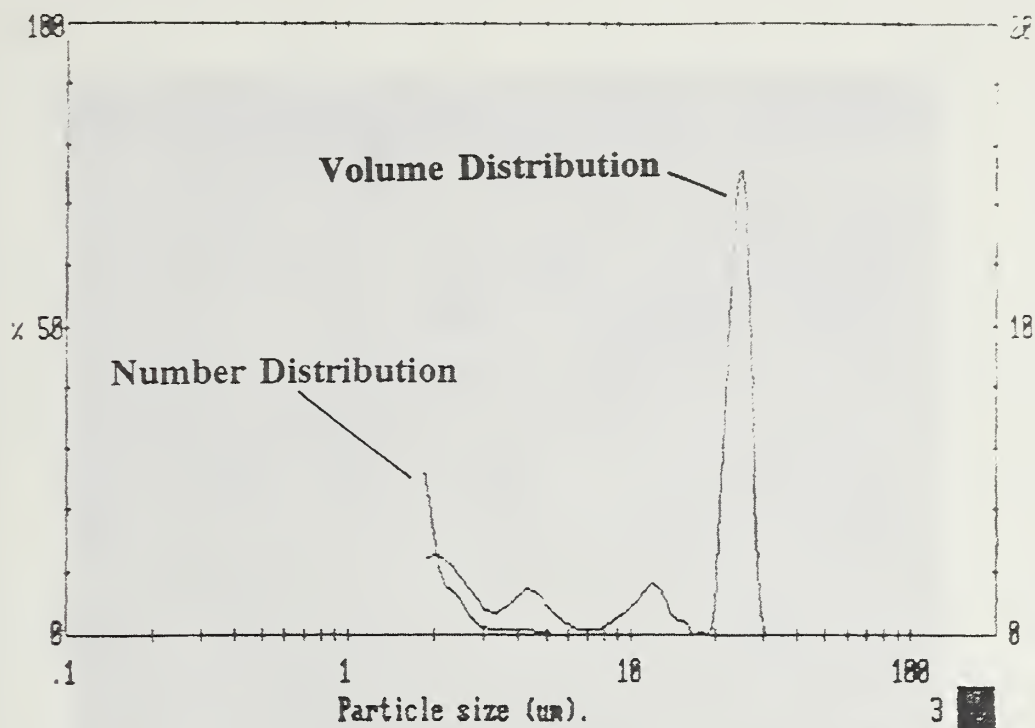


Figure 4.10 Quenched Malvern 2600 Plume Results

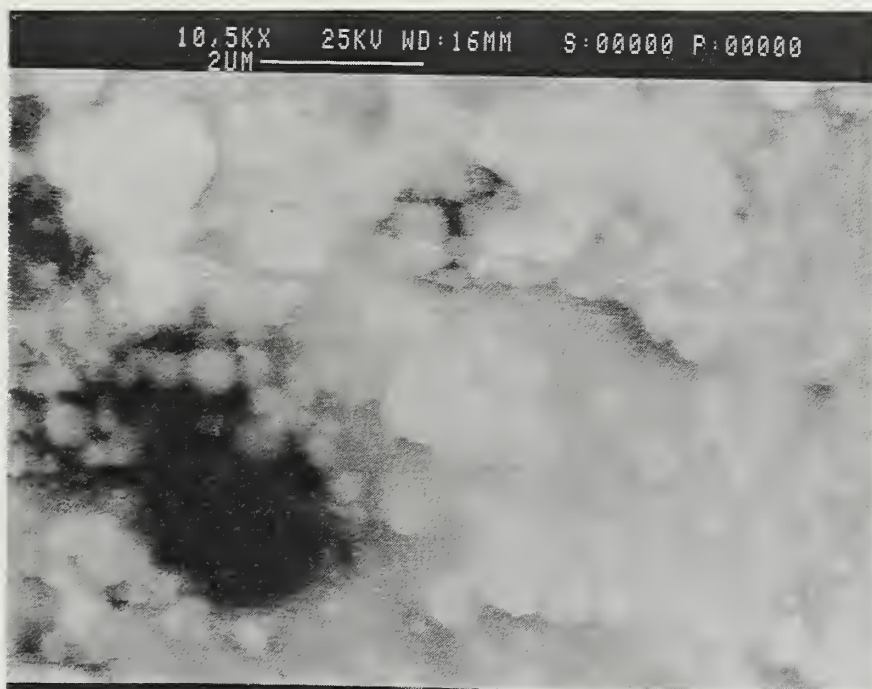


Figure 4.11 Quenched Impact Probe Sample



Figure 4.12 Quenched Impact Probe Crater

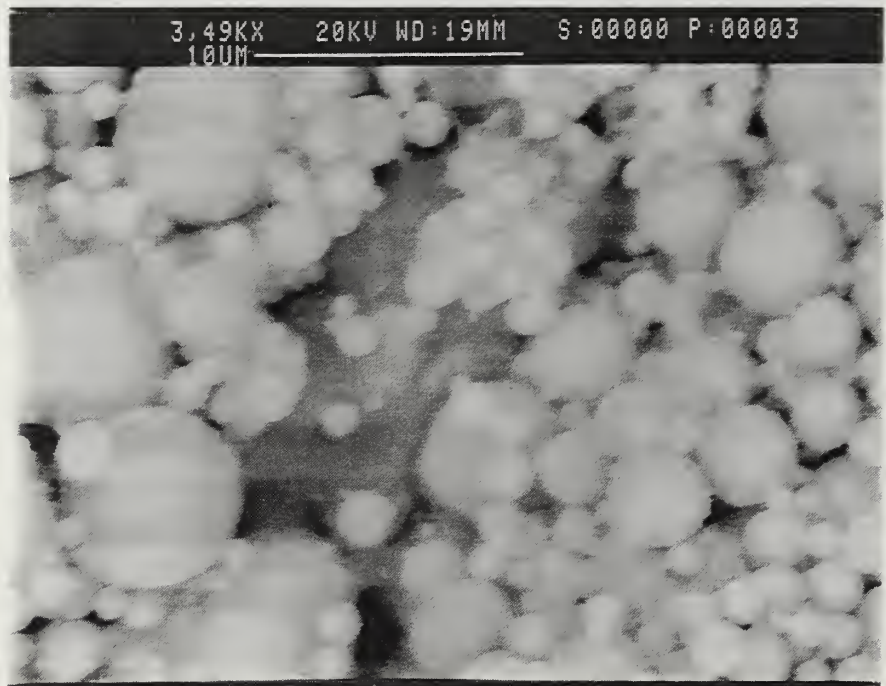


Figure 4.13 Quenched Nozzle Sample



Figure 4.14 Quenched Nozzle Sample Showing Cracked Shell

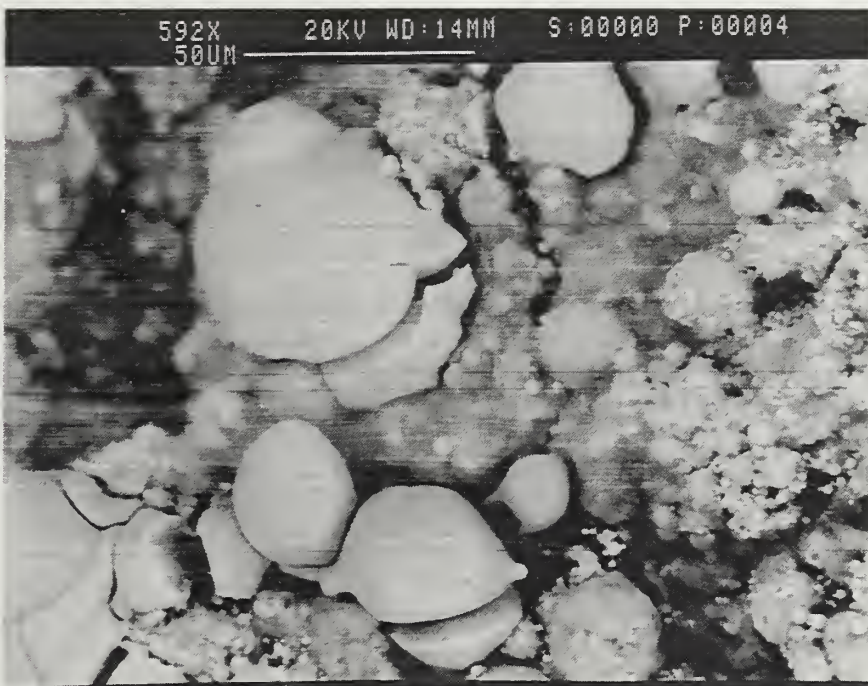


Figure 4.15 Quenched Nozzle: Silicon Based Particles

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